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Crystallinity for natural cellulose in jute fibre

KH. M. MANNAN AND M REAZUDDIN

Physics Department, Dacca University, Dacca-2, Bangladesh

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Percentage of crystallinity for different varieties of jute fibre has been determined by using an X-ray diffraction theory based on order-disorder model Correlation coefficients between crystallinity and breaking stress on this and older methods are 0·74 and 0·45 respectively indicating more reliability of the method reported here

1. INTRODUCTION

Crystallinity together with the molecular weight and orientation are the most important factors in determining the mechanical properties of polymers. The paper and jute industries have needed a correlation between crystallinity and texture of wood pulps and jute fibres. A number of methods for estimating disordered and crystalline fraction of the cellulose based on X-ray diffraction or reaction rate studies have been reported. Conard & Scroggie (1945) measured percentage crystallinity from X-ray patterns as

$$X_{Cr} = \frac{(I_1 - I_m)}{I_1} \times 100. \qquad \ldots (1)$$

where I_1 is the average intensity from the (101) interference peak and I_m is the background intensity between (101) and (101) interference peaks

Clark & Terford (1955) modifies this by taking the ratio of the (002) interference peak arising from the crystalline aggregate to the intensity of the amorphous halo which occurs between ($10\bar{1}$) and (120) reflections. However, the correlation coefficient of 0·24 between percentage crystallinity and breaking stress for paper pulp was quite low indeed

Recently Ruland (1961) has developed a diffraction theory for fibres based on order-disorder phenomenon. There are infinite number of possible arrangements of the molecular chains of cellulose molecules and two limiting cases require particular attention The first can be thought of as a completely isotropic and amorphous aggregation of randomly oriented and kinked chains (*disordered or amorphous region*); the second a state of perfect 3-dimensional order and orientation in which all the chains lie parallel on a regular spatial lattice (*crystalline region*). In a real cellulose fibre there is a continuous transition

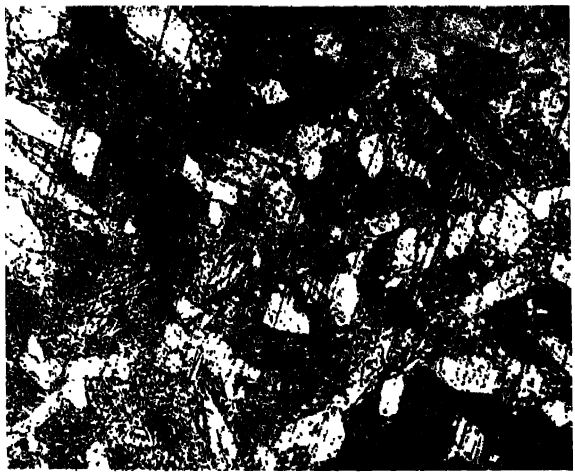

Fig. 1

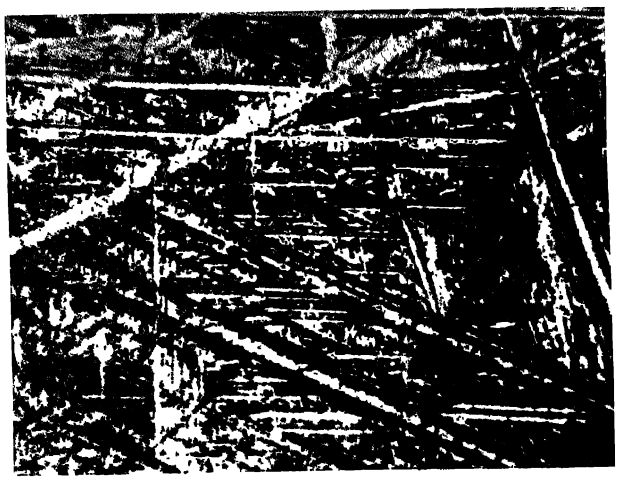

Fig. 2

Fig 3

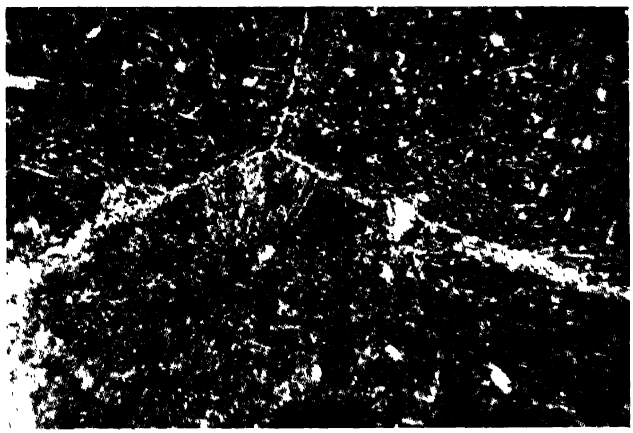

Fig 4

Fig 5

from one region to another With this model Ruland defines percentage of crystallinity as

$$X_{cr} = \frac{\int_{S_0}^{S_p} S^2 I_{cr}(S) \partial S}{\int_{S_0}^{S_p} S^2 I(S) \partial S} K(S_0, S_p, D, f^2) \quad \ldots (2)$$

and

$$K = \frac{\int_{S_0}^{S_p} S^2 f^2 \partial S}{\int_{S_0}^{S_p} S^2 f^2 D \partial S} \quad (3)$$

where, the reciprocal vector S is given by $2 \sin \theta/\lambda$ corresponding to the Bragg angle θ; $I(S)$ is the intensity from the amorphous portion at the end of the reciprocal lattice vector S ; I_{cr} (S) is the intensity from the crystalline part of the fibre at the end of S; D is a disorder function, and S_0 and S_p are reasonable limits of integration

2 Procedure

For each sample a graph was plotted with IS^2 along Y and S along X-axes respectively. A separation line from tail to tail separates intensity due to the crystalline region from the background intensity due to the amorphous region.

The area under peak gave $\int_{S_0}^{S_p} S^2 I_{cr}\, ds$ and that below the straight line gave $\int_{S_0}^{S_p} S^2 I\, ds$ where S_0 (2θ 10°) and S_p (2θ 45°) were so chosen that intensity was mainly from the background.

The disorder function D is given by

$$D = \exp(-K'S^2). \qquad \ldots (4)$$

For native cellulose $K' = 6.0$ accounts for lattice imperfections and thermal vibration

$K = 1{\cdot}2$ was found graphically. Atomic scattering factors used were those from the International Tables for X-ray Crystallography (1962).

3 Experimental

3.1 *X-ray study*

Input power supply was stabilized by an electronic voltage stabilizer The specimens of jute samples were made by cutting them into thin slices along their lengths and were taken into the sample holder in such a way that the specimen thickness was uniform Data were collected automatically by a diffractometer which was calibrated by Al and Si standards The diffracted Cu$K\bar{\alpha}$ patterns were monochromatized for $K\alpha_1$ by a curved quartz crystal which also act as a focalizer

3.2 *Measurement of Breaking Stress*

A chemical balance was specially modified for the measurement of breaking stress of single fibres (Hafiz & Mannan 1970) One of the pans was replaced by an equal weight at the end of which the fibre could be fixed in a slit which closes down by a screw. The other end of the fibre was fixed in a similar slit, the entire thing at the bottom could be moved up or down by the movement of a large screw. The movements could be measured by a linear scale and vernier Thus weights required to balance the stram upto the point the fibres break were recorded. The average diameter of each variety was determined by a travelling microscope.

4. Results

4.1 Percentage of crystallinity and breaking stress for different varieties of jute fibre are given in tables 1 and 2 respectively.

Table 1 Percentage of crystallinity ($K = 1{\cdot}2$)

Specimen	Area for amorphous region. $\int_{s_0}^{s_p} S^2 I(S)ds = a$ in arbitrary unit	Area due to crystalline portion $\int_{s_0}^{s_p} S^2 Icr(S)da = b$ in arbitrary unit	Percentage of Crystallinity $Xcr = \frac{Ka}{b} \times 100$
Tosa *A*	53·5	98·5	65·0
Tosa *B*	47·5	103·2	55·0
White Special	49 2	113·3	52·0
White *A*	59·5	138·5	51·5
White *B*	45·2	111·3	48 7
Mesta *A*	41·8	106·4	47 0
Mesta *B*	37·5	104·3	43 0

Table 2. Breaking stress

Specimen	Breaking load w in gm.	Average Radius r in cm.	Breaking Stress Y in Kg/cm^2.
Tosa *A*	126	0 0050	1605
Tosa *B*	91	0·0046	1374
White Special	59	0·0038	1305
White *A*	53	0·0037	1292
White *B*	78	0·0045	1228
Mesta *A*	81	0·0047	1169
Mesta *B*	66	0·0044	1086

4 2 Correlation coefficient (r) between crystallinity and breaking stress for different varieties of jute fibre calculates as 0·74 which is an improvement over the value of 0·45 for those specimens when percentage of crystallinity was calculated using eq. 1 (table 3).

Table 3 Percentage crystallinity using eq. (1)

Specimen	Average intensity from (101) peak $= I_1$	Background intensity between (101) and (10$\bar{1}$) peaks $= I_m$	$Xc_r = \frac{I_1 - I_m}{I_1} \times 100$ % Cryst.
Tosa *A*	35	18	48·5
Tosa *B*	39	25	38·6
White Special	38	25	34·2
White *A*	48	25	27·3
White *B*	37	28	24·5
Mesta *A*	37	39	21·6
Mesta *B*	36	30	16·6

5 Conclusion

Clark & Terford (1955) had reported a correlation coefficient of 0·24 for different varieties of paper pulp. Khan (1971) had found r-values of 0·50 and 0·86 using the old and Ruland's (1961) theories respectively for different varieties of manufactured papers. The present value of 0·74 for r using eq (2) in comparison with 0·45 using old theory speaks much in favour of Ruland's theory (1961).

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